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Trace element analysis of hot spring water by Particle Induced X-ray Emission (PIXE) method

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Abstract : Particle Induced X-ray Emission (PIXE) technique is well suited for analysis of natural water due to its multielemental capability (Na to U) and uniform sensitivity The high absolute sensitivity of PIXE, makes it possible to perform analysis of very small size samples [1] Proton beams of 2–4 MeV obtained from the low energy tandem pelletron accelerator at Institute of Physics are used for the trace element analysis of water. Experimental facility and sample preparation laboratory have been established for PIXE analysis of environmental samples PIXE method in combination with preconcentration technique, very low detection limits have been obtained for trace element analysis of hot spring water Sample collection, preparation, irradiation, experimental setup and spectrum evaluation are discussed

Keywords : Trace element analysis, water, PIXE

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PIXE, the technique used is a variant of the broad family of X-ray emission techniques. Heavy charged particles (typically 2–4 MeV of proton) are used to produce the characteristic X-rays from the analyte elements in the sample. The combination of PIXE with Rutherford Back Scattering (RBS) and Proton Induced Gamma Emission (PIGE) spectroscopy, in order to extend analytical capabilities to almost the whole periodic table has been already analysed [2]. Usually, in such application RBS and PIGE are used for detection of light elements, while PIXE is used for the determination of heavier elements ($Z > 15$).

Natural water in its different forms have been interesting materials of study for trace element analysis in relation to pollution and environmental hazards. Atri and Taptapani are the two natural hot springs of Orissa located about 40 kms and 200 kms respectively from Bhubaneswar. Water samples collected from these natural sources are analysed to study their elemental concentrations. The low level metal concentrations in water samples makes the sample preparation technique a challenging job for PIXE analysis. Suitable precipitation

techniques and coprecipitation procedures have been adopted for best possible analytical results.

Precipitation as a sample preparation technique is shown to be simple, relatively rapid and accurate for trace element preconcentration from natural water [3]. Considering the low metal levels in natural waters, preconcentration method has been used for the water sample preparation for coprecipitation of dissolved metals as carbamates. The glass wares and allied apparatus used are all borosilicate glass. Initially apparatus are washed using a mixture of hydrochloric acid and nitric acid and they are finally rinsed with the ultra pure (Millipore) water of resistivity 17 MOhm/cm. The saturated sodium diethyldithiocarbamate (NaDDTC) solution is prepared freshly before each experiment. 100 ml of water sample has been taken for coprecipitation of dissolved metals in the sample. 100 μg of palladium (Pd) is added into the water as coprecipitant and internal standard. The pH is increased to 9 by adding ammonia (NH_3) and 1 ml of NaDDTC solution is added to precipitate the presence of elements in the water sample as carbamates. Palladium diethyldithiocarbamate is also formed in the intermediate reaction, which is a quite stable complex in the water and acts like a good coprecipitating agent [4]. The precipitate thus formed is filtered and transferred on the Nuclepore polycarbonate membrane filter (pore size 0.2 μm diameter). The Nuclepore filter and the sample layer on the filter membrane form a thin target. Filtration efficiency of filter is evaluated with a double filtration technique. This gives 99% efficiency for the metal under consideration. A NIST reference water (SRM 1643) is also prepared with the same procedure.

PIXE measurements have been carried out at the 3 MV pelletron accelerator (Figure 1) at Institute of Physics using the equipment and measurement condition previously

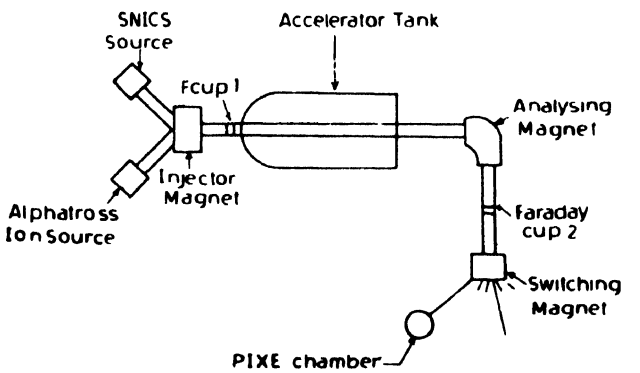

Figure 1. Schematic diagram of the accelerator

described with details [5]. A cesium sputtered negative ion source (SNICS) is being used to get the 3 MeV proton beam. Singly charged (H^-) negative ions from a TiH_2 cathode at 2 to 4 KV are preaccelerated at 67.5 KV and then led to the main accelerating tank by an injector magnet. The accelerated ions are stripped at the midterminal through a charge exchange process with a N_2 gas stripper system. After emerging from the accelerating tank

the ion beam is energy/momentum analysed by a 90° analysing bending magnet and then directed to the scattering/PIXE chamber kept in vacuum (~ 10^{-6} Torr) through a switching magnet.

Sample targets are mounted in a target ladder. Then the target ladder is loaded into the PIXE chamber and the irradiation is carried out in the vacuum conditions. The sample targets are kept in PIXE chamber at 45° to the beam and seen by the Si(Li) detector at 90° to the beam. The targets are exposed to a 3 MeV proton beam and beam current is in the range to 30–40 nA. The characteristic X-rays from the sample are collected by the energy dispersive detector Si(Li) which is having an energy resolution of 165 eV at 5.9 keV. Spectrum is recorded by using a Canberra series 88 multiparameter analyser. PIXE spectra of Atri and Taptapani hot spring water samples are shown in Figures 2 and 3. Spectral data is transferred to a computer where the analysis is done further.

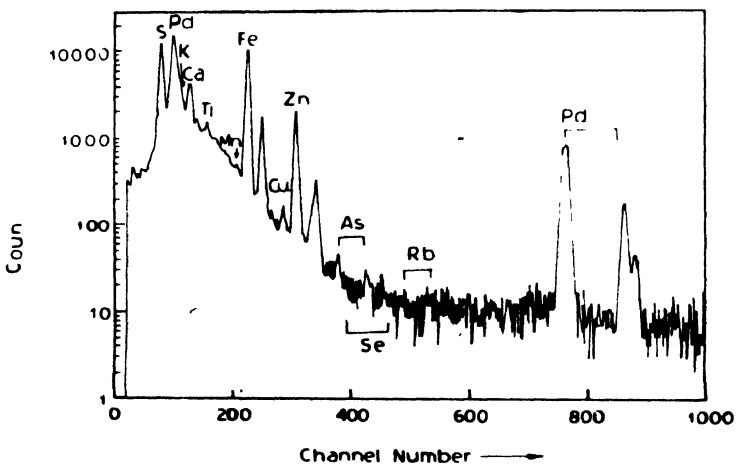

Figure 2. PIXE spectrum of water from hot spring at Atri.

The spectrum obtained from each sample is analysed after subtracting the appropriate background contribution from measurements of blank target. The number of characteristics X-ray of element in a thin target is proportional to the original concentration in the sample. X-ray intensities are calculated using the AXIL program (supplied by International Atomic Energy Agency) by the least square fitting method. The elemental concentrations are calculated as

$$C_x = (K_s / K_x)(I_x / I_s)C_s$$

where K, I and C are proportionality constant, X-ray intensity and concentration. The symbols x and s refer for element and internal standard, respectively. The parameter (K_s / K_x) for each element under observation is obtained by calibration made under the same experimental conditions (Figure 4).

In the precipitation procedure used for water sample analysis, contamination contributed due to impurities in the used chemical reagents has been measured using a

blank target. It is found that contamination introduced by the reagents is in small concentration. The elemental concentration levels of water samples from Atri and Taptapani

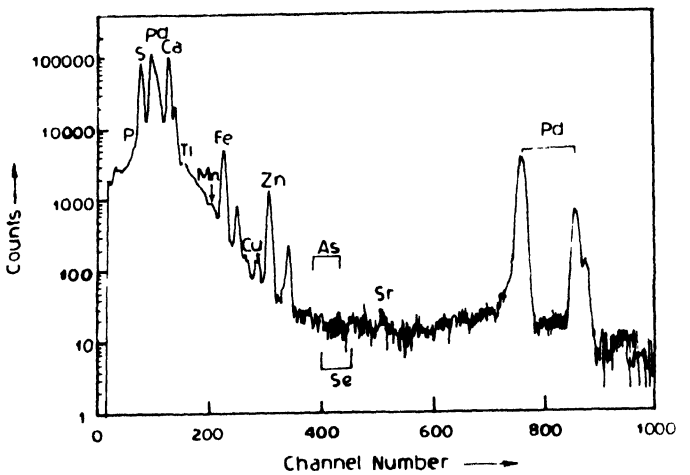

Figure 3. PIXE spectrum of Taptapani water

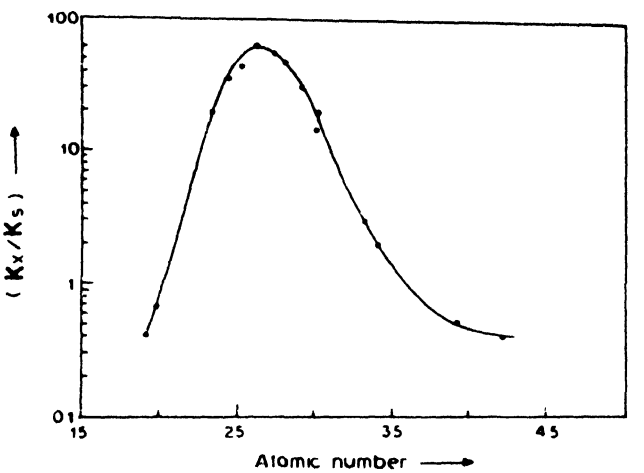

Figure 4. The variation of calibration factor with atomic number of the element

hotsprings are given in the Table 1. It is observed that the concentration levels of S, Ca and Fe are higher in Atri water sample than the Taptapani water. Levels of elements like Cr, Ni and Cu are found to be very low in both cases. Drinking water standards [6] as per World Health Organisation (WHO) are also presented in the above table. The elemental analysis of water samples from Atri and Taptapani natural hot springs using PIXE technique shows that the concentration levels of metals are below the maximum permissible limits of WHO.

A PIXE technique is shown as capable of detecting low concentrations of metals in natural waters. PIXE is a promising analytical tool and its availability at

Institute of Physics opens the avenues for a variety of environmental analysis in near future.

Table 1. Elemental concentrations (μg/l) in drinking water samples from different locations and WHO standards

Element	Atri	Taptapani	WHO standards	
			Permissive	Excessive
K	1.0×10^3	2.3×10^3	10×10^3	12×10^3
Ca	47.6×10^3	16.6×10^3	75×10^3	200×10^3
Cr	2.1	1.6	50	-
Mn	10.4	44.7	50	500
Fe	185.2	127.6	300	1000
Ni	20.2	14.9		-
Co	9.4	-	-	-
Cu	89.1	25.0	1000	1500
Zn	75.5	62.7	5000	15×10^3
As	4.4	6.1	50	-
Pb	1.4	3.1	50	-

Acknowledgments

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